TECHNICAL APPLICATIONS FOR OXIDE-DISPERSION STRENGTHENED MATERIALS

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G. Mervin Ault* and H. M. Burte**

INTRODUCTION

It seems appropriate to introduce a symposium on oxide-dispersion strengthened materials with an attempt to outline potential applications for this new class of materials and, indeed, this is our primary purpose. Although it is impossible to list all the components of all the hardware that may utilize such materials, we have attempted to describe examples that will illuminate problem areas and aid us in indicating credible development goals for oxide-dispersion strengthened materials. Clearly there are many controversial and poorly defined aspects of the processing and use of these materials. As an additional objective, we wish to stimulate discussion that may define and perhaps resolve some of these controversial points and that may define the more significant areas and approaches for both fundamental study and materials development.

Before discussing potential applications for these materials, it is necessary to answer the question: what unique characteristics do they offer? The primary interest in dispersion strengthened materials is based, of course, on their stability at very high temperatures, as shown in Figure 1. Two comparisons are shown: nickel plus a dispersed oxide of thorium (1) compared to an outstanding wrought nickel alloy, and aluminum plus a dispersed oxide of aluminum (2) to a high temperature aluminum alloy. They are compared on the basis of their 1000-hour rupture strength at various absolute temperatures expressed as a fraction of the melting point of the base metal. In both cases,

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although the dispersion strengthened pure metal is weaker than the precipitate-strengthened solid-solution matrix alloy at low temperatures, the dispersion strengthened materials are less sensitive to increase in temperature and thus are superior in strength above 0.55 to 0.65 of the melting point of the pure metal. This is the most important characteristic of inert particle-dispersion strengthened metal systems: strength and stability to unusually high temperatures.

The presence of the stable hard particles aids strength in two ways. First, they are very stable at very high temperatures, and stay in the matrix at the proper size and spacing to impede creep at temperatures where precipitated particles would agglomerate or dissolve. Second, the presence of stable particles permits development of perhaps a unique cold-work strengthening and favorable structures during thermo-mechanical processing, and permits retention of these processing benefits to unusually high temperatures. This then defines the first and major area of interest for oxide-dispersion materials: As a general rule and at the present state-of-the-art, oxide-dispersion strengthened materials would be considered whenever it is necessary to extend the use of a particular alloy system or matrix to a higher temperature than can be achieved by conventional strengthening methods.

Two aspects of this statement warrant emphasis here and will be illustrated and evaluated in more detail later. The first is "at the present state-of-the-art" which involves questions of cost and the additivity of strengthening mechanisms. The second is "necessary" and involves the competition between a particular alloy system and higher temperature competitors such as aluminum versus titanium.

Another advantage associated with dispersion strengthening is that the

melting point of the matrix is not compromised. Solid solutions can have sig-
nificantly lower melting points than the pure element, and in complex alloys
minor phases that melt at even much lower temperatures can occur. Examples are
shown in Table I. Retention of melting point is important in applications
where the materials are used near their melting point, such as in the nozzle
of a solid propellant rocket, and where momentary over-temperatures may occur
as for components near the combustion zone of turbine or ramjet engines.

Another potential advantage is that dispersion-strengthened pure metals
will have a higher thermal and electrical conductivity than the same metal
when solid solution strengthened. Thus, dispersion-strengthened materials may
find application where both strength and thermal conductivity are required, e.g.,
for components of heat exchanger parts including nozzles of regeneratively
cooled rockets, or in components where an improved thermal fatigue resistance
may be achieved through lowering of thermal gradients. The electrical conduc-
tivity advantage would be useful in electrical conductors or electrical contacts.

Having considered the general nature of dispersion strengthening it is
appropriate to inquire about those directions for future effort which might prove
to be most rewarding from the viewpoint of eventual application and use. We
will tend to focus attention upon a time frame of 2 to 12 years hence, and to
provide illustrations with aerospace applications with which we are most familiar.
It is essential that such an analysis and projection be accomplished within a
framework of active competition between different materials and approaches. Un-
wise selection and achievement of development goals which do not provide an im-
provement over capabilities of competitive available materials can lead to the
sense of futility and frustration which was sometimes experienced in the early
major materials development areas following World War II.

There are few, if any specific routes for materials development which offer the only, or even the "clear cut" best possibility of satisfying future technological requirements. The simple unambiguous requirement -- such as for an alloy with tensile strength X, and ductility Y at temperature Z, which will definitely find use if achieved -- rarely exists. There are usually competing materials approaches such as precipitation hardening stainless steels versus titanium alloys. Even more important from the viewpoint of expanding the scope of possibilities which must be considered, there are always a variety of hardware or design approaches which might be taken to provide acceptable overall system performance. Thus, the problem of heat might be solved by convective cooling, radiation cooling, or ablation. Each of these will suggest its own spectrum of materials approaches. Cost will, of course, play a role, and include not only the basic cost of the material but also the costs of fabricating it into the desired configuration and assembly into the final hardware. In a comparison between titanium and aluminum the cost of machining a given configuration may be more important than the price per pound of the alloys involved. It is also obvious that more than one property of a material must usually be considered, such as ductility of weldments or oxidation resistance, in addition to strength. This provides yet another dimension to the analysis. The final selection will, of course, be on the basis of the overall cost-effectiveness of different engineering approaches. This may be relatively insensitive to the choice of materials and thus provide little stimulus to future materials research and development, or it may show that even very expensive materials can effect significant systems savings as do high strength materials for the upper stages of a rocket. The difficulty of attempting to identify fruitful directions for materials development is clear. The extent of uncertainty which this complex

competition introduces into the tentative conclusions that can be drawn at any point in time must always be appreciated. The need for extensive information about candidate competitive materials to permit them to be effectively evaluated should also be obvious.

Much of the preceding discussion will be pertinent to and reflected in the analysis of possible materials development objectives and opportunities for application of dispersion strengthening. The current high costs of new or developmental dispersion strengthened materials will undoubtedly decline with production experience and/or greater quantity use. However, there is not now any reason to believe that they will provide a low-cost panacea. It is suggested that the best opportunity for successful competition and first generation application of dispersion strengthened materials will be in critical areas where alternate conventional materials suffer serious shortcomings. We believe, for example, that it will be clear that utilizing dispersion strengthening to extend the temperature range of availability of strong ductile oxidation-resistant materials into the range from 1800 to 2500° F provides one of the best opportunities for successful competition.

DEVELOPMENT GOALS

Let us now consider the metal or alloy systems to which dispersion strengthening might be applied and include comparisons with existing materials or other routes toward improving materials properties. Such comparison will often involve a strength to density ratio. The opportunity to reduce the weight of and therefore increase the performance and efficiency of aerospace systems is, of course, related to such ratios. This is well recognized as one of the most profitable goals for materials development and provides an appropriate starting point for the discussions.

As we increase temperature, if one alloy system will not achieve the needed strength we generally substitute an alternate higher temperature system. If aluminum alloys cannot perform the needed function we turn to titanium or steel alloys; if nickel alloys are unsatisfactory we turn to refractory metals. It is important, however, to recognize an oxidation barrier in the temperature range of 1800 to 2000° F. Below this temperature strong "ductile" oxidation-resistant alloys are available. Above this range: conventional oxidation-resistant metals become too weak for most uses; current refractory metals suffer from catastrophic oxidation and must be protected with coatings; strong oxidation resistant materials, such as intermetallics and ceramics, are brittle. The gains which very high-temperature materials can offer has stimulated much activity and progress in coated refractory metals, or the use of brittle materials, but the problems are many, reemphasizing the potential for application of dispersion strengthening to oxidation-resistant matrices. Certainly, at lower temperatures, and we shall discuss the entire temperature range, the existence of other materials approaches will increase the behavior increment which a user will demand before he will seriously consider a new material, and this should be reflected by adequately advancing the goals for dispersion-strengthened materials.

Aluminum, Titanium and Steel

At the lower end of the temperature range, from ambient to about 1200° F, efficient and _relatively_ well-characterized and understood structural materials are available. Figure 2 shows the yield strength to density ratios for both well-established and experimental materials. This parameter based on yield strength is commonly used as an initial indication of the relative structural efficiency of these materials particularly at low fractions of the melting point.

At higher temperatures, for a specific alloy system, this would not provide an adequate comparison for anything except very short time uses and creep and rupture properties must be considered. Rupture strength data for some established materials for use at higher temperatures are shown in Figure 3 (3, 4). Also, of course, for specific applications, other properties such as impact toughness or fatigue strength may be controlling, and the order of merit between different materials is by no means the same when using these different properties.

The full line curves in Figure 2 are for well-established materials. An aluminum alloy (2024-T86); a titanium alloy (Ti-6Al-4V, annealed); a precipitation hardening stainless steel (PH 15-7Mo, TH 1050); and a nickel-base super-alloy (René 41, annealed) are shown as representative of this group. The data given (5) are conservative values suitable for use in design. A broad variety of other information such as data for the other properties mentioned above as well as experience in and developed procedures for fabricating these materials into a wide variety of configurations are also available. For many applications it is the buckling resistance or stiffness of the material and structure which controls and not necessarily the tensile properties we have been discussing, and then the ability to fabricate a stiffened structure such as a honeycomb sandwich becomes quite important. The acquisition of such a wide range of property information and fabrication capability is obviously a major undertaking involving much time and resources. The availability of extensive property information for an older better-established material can, at a given time, be a deciding factor in not selecting a newer perhaps inherently more efficient material for a given application. Obviously, expenditure of the considerable effort required to acquire the broad range of experience necessary to support

the eventual selection and use of a new material will be warranted only if the initial indications of the improvement increments, as measured by relatively simply acquired properties such as yield strength to density ratio, are sufficiently large.

Even within the well-established structural materials there is a considerable element of competition. Thus, there is much current controversy relative to the use of the highest strength, most structurally efficient aluminum alloys or tempers versus using somewhat lower strength with the advantage of higher corrosion or stress corrosion resistance; the use of the T6 versus T73 tempers of 7075 aluminum alloy is an example. As another example, Inconel 718 is shown in Figure 3 to be equal to or slightly stronger than René 41 up to 1300° F. At the low end of the temperature range given in Figure 3 (near 1200° F) Inconel 718 might be a more desirable choice for many applications because it is more easily welded and forged. At somewhat higher temperatures (1400° F), René 41 is stronger and its use is more clearly indicated. On the other hand, for applications where weight saving is not critical, the lower cost of the weaker low-nickel alloys, such as A286 or stainless steel, would indicate their use.

Points are also shown in Figure 2 for a variety of less well-established materials including both "conventional" alloys and the more recent composite material approaches to illustrate the nature of the future development possibilities with which dispersion strengthening must compete. A point is shown for a heat treated condition of the Ti-6Al-4V alloy (6); there is less experience in the use of these conditions than in the annealed condition and they are generally less ductile. AFC 77 (7) is a high temperature corrosion resistant steel available in evaluation quantities. The point for Ti200X (8) repre-

sents an experimental laboratory lot of material in an alloy system initially explored by Margolin. The Ti-Bialloy (9) is a particulate composite of titanium at a similar stage of development as the Ti200X, whereas the point for the boron fiber-aluminum matrix composite (10) merely reflects the ability to combine this fiber and matrix without excessive destructive interaction. This latter point is for a unidirectional composite with 35 volume percent of continuous filament boron in an aluminum matrix. It must be emphasized that although the point shown does provide an indication of the possibilities in the composite approach it does not necessarily reflect the load carrying capability of the materials and structures which may eventually be developed.

Let us now compare a dispersion strengthened aluminum containing 12 percent Al_2O_3, S.A.P. (2), with the previously described existing capability and future possibilities which used other than dispersion strengthening. It is clear that although the ability of dispersion strengthening to maintain some strength in aluminum to quite high fractions of the melting point is indeed striking; it does not provide a material which is competitive with existing capabilities in other material for a wide variety of aerospace applications. There may be applications, however, where a property of dispersion-strengthened aluminum such as the inherent corrosion resistance to a specific environment or nuclear properties is a potentially overriding feature as in cladding for reactor fuel elements (11). There are also applications where cost may become much more important than weight saving as in non-flying systems. The possibility, for example, of providing increased high temperature properties to stainless steels which are low cost relative to high nickel or cobalt content alloys might be considered. (Such materials may be of interest to automotive gas turbines.)

It seems that in the temperature range up to about 1500° F, (approximately

0.6 M.P. of Ni) and for applications where strength-to-weight ratio is
the major criterion, dispersion strengthening should be combined with other
strengthening mechanisms if a competitive material is to result. Even major
improvements in the inherent ability of dispersion strengthening to strengthen
a pure metal at elevated temperatures, such as increasing from I to II in
Figure 4 is unlikely to be adequate to provide a competitive material in this
temperature range because other materials are available. One must restate
the familiar question of the extent to which dispersion strengthening can be
added to other strengthening mechanisms. We know that it can provide elevated
temperature strength as represented by curves I and possibly II. Will it in
general achieve this at the expense of strength at lower temperature (Curve I),
strengthen only at high temperatures (Curve III), or can it provide a strength-
ening increment over a broad range of temperatures (Curve IV)? The answer is,
of course, not simple. Degradative interactions between the dispersoid and
other alloy constituents may occur and the problems of matching the most desir-
able thermomechanical histories for different strengthening mechanisms will be
formidable. The intent of the question is, however, valid. With increasing
possibilities of creating fine dispersions in alloy systems, increased attention
should be given to elucidate the possibilities of combining strengthening mech-
anisms as a means for achieving improved materials in this range of temperatures.

Superalloys and Refractory Metal Alloys

To extend the strength comparison into the high temperature range, from
just below to well above the oxidation barrier, we may refer to Figure 5. It
includes the high temperature superalloys of nickel or cobalt base and alloys
of the more refractory metals: chromium, columbium, molybdenum, tantalum, and
tungsten. Focus attention now on long time rupture strength. Density compen-

sated properties and short time properties will be considered later. The superalloys have been the materials most widely used at high temperatures. The refractory metal alloys shown here are the result of development activities conducted in the past five to seven years -- most were unknown prior to that time. The alloys on the figure are those reported to have about the highest rupture strength of each class. Although the alloys shown for Cb, Mo, and Ta are reasonably well advanced, experience with them is not yet comparable to lower temperature alloys. The lower strength Cb alloy (typically D43 or Cb752) and the Ta alloy (advanced T222 (12)) are quite ductile even in the recrystallized condition, and are readily welded and fabricated. The higher strength Cb alloy (13) and the Mo alloy, though fabricable, are not ductile when welded. The tungsten alloy is in the "laboratory" stage (14), would be processed with considerable difficulty, and is brittle at room temperature. The chromium alloy is a recent development (15), is in the "laboratory" stage, and processing problems are unexplored. The alloy, though strong, retains other short-comings of chromium. (Summaries of developments in refractory metal alloys may be found in references 16, 17, and 18).

If strength is the only criterion for material selection, this plot would indicate that as operating temperature is increased, we may successively change from nickel alloys to chromium, to columbium, to molybdenum, to tantalum, and to tungsten. On the basis of strength alone, there would not be an obvious need to attempt to achieve the benefits of dispersion strengthening in any of these materials except perhaps tungsten, but other factors, of course, lead to its further consideration.

There is obviously no higher temperature successor to tungsten, and dispersion strengthening of it will be a route to the highest possible temperature

capability with metals. Sell and associates (19) of Westinghouse Lamp Division have been conducting studies on oxide dispersion strengthening of tungsten. Their best alloy to date is tungsten - 3.8 v/o ThO_2 containing a high concentration of ThO_2 particles of less than 500 A$^\circ$ size. The alloy is reported to show unusual structural stability to 5250° F or 0.87 of the melting point. The short-time tensile strength of this alloy compared to unalloyed tungsten and an arc-melted tungsten-hafnium-carbon alloy is shown in Figure 6. The W-Hf-C alloy (14) derives its strength in part from dispersed HfC particles and is significantly stronger than the tungsten-ThO_2 alloy at 3500° F (0.6 M.P.) even in the recrystallized condition. Data are not available at higher temperatures for the W-Hf-C alloy where the carbide phase may be unstable and where the oxide-dispersion-strengthened material would be expected to show superiority. At temperatures near 0.8 of the melting point of W this W-ThO_2 alloy has about four times the strength of unalloyed W and appropriate W-ThO_2 alloys may retain useful strength to a maximum of 5500° F (the melting point of ThO_2). The advantage of the W-ThO_2 alloy would be expected to be even greater in creep or stress-rupture at the higher temperatures, but such data are not yet available.

Although it is appropriate to explore the ultimate potential for metal systems with dispersion strengthened tungsten alloys as is being successfully accomplished in the Westinghouse program, the need for these materials does not at this time seem great enough to warrant major emphasis. There are, at present, few applications where strength at such high temperatures is sought. One may be in the nozzles of solid propellant rocket motors, another is in furnace heater elements or radiation shields where the strength and particularly the unusual high temperature stability to minimize recrystallization and concomitant low temperature embrittlement of such materials may be an advantage.

Improved Ductility with Dispersions

An important factor, beyond that of strength alone, that must be considered is ductility. The body-centered-cubic metals including iron and the important refractory metals chromium, columbium, molybdenum, tantalum, and tungsten exhibit a transition from ductile to brittle behavior as the temperature is lowered. The ductile to brittle transition temperature (DBTT) at which this occurs is a complex function of purity, alloying, prior thermomechanical processing history, test method and conditions, and the specific test used for measurement of ductility. The alloys shown in Figure 5 for Cb, Mo, and Ta are all ductile at room temperature in conventional tensile or bend tests. Under the same conditions the high strength chromium and tungsten alloys on this figure have a DBTT well above room temperature as do high strength recrystallized molybdenum alloys in some cases. It is a continuing goal in research with all of the refractory metals and particularly in tungsten and chromium to find methods or alloys that will combine maximum strength with minimum DBTT.

It is accepted that properly distributed oxide particles may retain strength to higher temperatures. Some evidence can be presented to suggest the intriguing possibility that particles, if properly distributed, can improve ductility. Rosenfield and Hahn (20) have conducted an analysis of the effect of second-phase particles on ductility and present arguments that suggest that under some conditions ductility can be improved, and DBTT lowered, by second-phase particles. To experimentally verify their predictions, they have conducted tensile tests at 77° K on iron and iron with dispersed Al_2O_3. They showed that the iron with dispersed Al_2O_3 was ductile whereas the iron without Al_2O_3 was brittle. In addition, the iron with Al_2O_3 supported almost twice the stress. Sell and co-

workers in their work on tungsten - 3.8 V/o ThO_2 alloys previously described report that the DBTT of their material after annealing at 2400° C was 190° C and thus was much lower than unalloyed tungsten (though still well above room temperature). Earlier, Maykuth and co-workers (21) reported that ThO_2 as well as other oxide additions reduced the tensile DBTT of tungsten and when ThO_2 was added to a tungsten alloy of 5 percent Re, they found tensile ductility at room temperature. Some work by Ryan and Johnstone (22) suggests that similar effects may also be achieved by particles in chromium.

This potential for lowering DBTT while improving high temperature strength warrants further study, particularly in tungsten, chromium, and recrystallized molybdenum. Some factors of concern in relation to ductility of dispersion-strengthened metals will be discussed later, however.

Dispersion Strengthened Oxidation Resistant Alloys

Another and a very important factor suggesting the use of dispersion strengthening is oxidation resistance. We have previously offered our opinion that the prime current potential for dispersion strengthening is to extend the temperature range of availability of strong ductile oxidation resistant materials into the range from 1800° to 2500° F. If adequate properties can be developed, such materials might find extensive use in a broad variety of future turbojets and ramjets, and hypersonic and reentry vehicles. The relative oxidation resistance of the materials we have been considering is shown in Figure 7 (23 to 27). This figure shows the oxidation rate on the basis of weight change in $\mu g/cm^2/hr$ in the first hour. (That these are very short time data must be emphasized. The refractory metals oxidize catastrophically and long time data are not available. On the other hand, TD-Ni, chromium, and Nichrome generate protective scales and the one-hour data represent maximum rates before the protective scales have de-

veloped to sufficient thickness to be of major benefit.)

Chromium, although its scale is somewhat protective, has a higher oxidation rate than Nichrome (representative of the more oxidation resistant super-alloys) at the temperature range of interest. In addition, the chromium alloys suffer severe embrittlement when exposed to air as a result of nitrogen absorption. The possibility of providing them with a useful combination of strength and lowered DBTT has been previously considered, and these together with coatings to inhibit oxidation and nitrification provide an approach which continues to receive attention. The refractory metals oxidize at rates of nearly four to seven orders of magnitude greater than superalloys. Although alloying of the refractory metals reduces the rate of oxidation, none of the high strength alloys have sufficient oxidation to permit their use for even short times in air. This has prompted the development of a technology of oxidation resistant coatings which have considerable promise for short-life uses such as reentry vehicles but are far from adequate for uses such as a reliable long lifetime engine for a supersonic transport. The more oxidation-resistant refractory metal alloy possibilities have received little attention in recent years since they were weak or brittle and would still require a coating. A re-examination of their potential, using dispersion strengthening as one approach to improving mechanical behavior may be warranted. Combination of such a less catastrophically oxidizing substrate with an appropriate coating might provide a sufficient reliability/lifetime increment for many additional applications.

Even more promising is the possibility of dispersion strengthening to provide adequate strength to nickel or a nickel or cobalt base alloy. Thoria dispersion strengthened nickel, even with no further alloying, is much more oxidation resistant than current refractory metal alloys (Fig. 7). Figure 8 (30)

compares the more oxidation resistant materials on the basis of total weight change in 100 hours at the temperatures of interest. (In the 100 hours, these specimens are cycled to room temperature several times.) On the basis of these data most of these materials are limited to temperatures near 1900° F for long time uncoated use. TD-NiC (Ni - 20Cr - 2 ThO_2), a recent development of Dupont (31), shows significant improvement in oxidation resistance over other similar materials, however. Even if coatings are required to provide adequate oxidation or sulfidation resistance for specific applications, the resulting metal coating system will be considerably more reliable than the current coated refractory metals.

The conventional cast and wrought superalloys have been studied since World War II and are the highest-strength oxidation-resistant metal systems available for use as structural materials. Improvements are needed, but, based on past history and the present advanced state of development, large increases in maximum use temperature for conventionally strengthened superalloys is unlikely. Evolutionary development since World War II has raised the maximum allowable use temperature (on the basis of a 1000 hour rupture strength of 20,000 psi) from about 1400° to about 1800° F, or 20° F per year. This allowable use temperature is already very high, perhaps 0.75 of the melting point of the matrix and perhaps 0.85 of the melting point of the lowest melting phases and conventional strengthening approaches hold little further potential. Greater improvements appear to be possible with dispersion strengthening.

In the past two or three years new oxide-dispersion strengthened materials based on superalloys have been reported. The strength of two of these, Dupont's TD-Ni (1) and Sherritt-Gordon's DS Nickel (32) have been added to the data of Figure 5 and are shown as Figure 9. As mentioned earlier, these materials

have relatively low strength at low temperature but hold their strength to

higher temperatures and are thus superior to the conventional cast or wrought

superalloys at high temperatures. As shown here, they are superior in 1000

hour rupture strength above 1850° F. (Because of their great stability, if

comparisons were made for longer rupture lives, say 10,000 hours, they would

be superior to conventional alloys at even lower temperatures.) Attempts to

combine low and high temperature strengthening mechanisms are underway in

several laboratories. Dispersion strengthening of more oxidation resistant

bases, such as nickel-chromium alloys, are also underway. One example is

Dupont's TdNi (31).

<div align="center">Other Materials</div>

The potential for dispersion strengthening a wide variety of other

materials can also be briefly noted. Much currently available beryllium con-

tains significant quantities of BeO and might well be considered to be a dis-

persion strengthened material. There are many instances where corrosion resis-

tance for specific applications, or a unique property such as electric conduc-

tivity dictate the use of a given base material such as copper, silver, gold,

platinum or lead, and which might benefit from the increased high temperature

strength, or stability offered by dispersion strengthening. Dispersion

strengthened copper is a familiar example. The general concept of using in-

herently stable particles to provide stability and creep resistance at high

homologous temperatures might also have applicability to improving the proper-

ties of intermetallic compounds or ceramics. Finally, the possibilities of

combining dispersion strengthening with fiber or filament reinforcing might

well warrant attention. The strength of the metal matrix need not be high in

these cases, and the increment which dispersion strengthening can provide to

a pure metal at very high fractions of the melting point might be quite signi-
ficant. The possibilities of destructive interactions between the fibers and
dispersoids will, of course, have to be explored.

APPLICATION POSSIBILITIES

Let us further define promising directions for development of dispersion
strengthened materials, and illustrate problems to be overcome, by considering
some of the application possibilities which show near future potential. Through-
out the discussion one must remember: the need to consider many aspects of
materials behavior including fabricability and a variety of properties; the
variety of design approaches possible and how these influence the materials
competition; the types of problems which can arise with new materials and the
possible inapplicability of behavior criteria which are suitable for older
materials. One further word of caution is necessary. Even if a dispersion-
strengthened material proves to be the most efficient for a given design ap-
proach, and contributes toward a certain type of hardware or design by being
the most cost effective for a given mission, there is no assurance that produc-
tion and use will result. Broad conceptual changes of how to accomplish a
mission, such as ballistic missiles versus bombers, or in national goals or
the desires of the market place such as the exploration of space versus the
bottom of the ocean, can negate the analysis.

Turbojet Engines

The need for higher operating temperatures in turbojet engines has been
well advertised in recent years and presents a major opportunity for the use
of dispersion strengthened nickel or cobalt base alloys. Turbine inlet tem-
peratures have been limited by the survival capability of certain critical
engine components. Perhaps the most limiting components are the turbine stator

vanes that must withstand direct impingement by the high velocity gases exit-

ing from the combustion chamber (to redirect these gases against the rotating

buckets), and the highly stressed rotating turbine buckets. As the designer

has exhausted the capability of the materials available to him, he has turned

to the alternative of cooling. Cooling these components must be paid for by

increased engine complexity and by some sacrifice in performance, when com-

pared with the same temperatures if achieved without cooling. It therefore

remains an important objective of metallurgical research to find materials that

will survive at higher temperatures thus permitting higher gas temperatures--

either without cooling or at least with reduced cooling requirements. Other

critical areas where improved materials may find use are in the sheet com-

ponents of the combustor and afterburner.

To provide proper perspective for our review of these materials, it is

desirable to have a knowledge of the strength levels required for each com-

ponent. Let us first consider the rotating turbine bucket. (A typical bucket

is shown in Figure 10.) In an uncooled bucket there is a "critical zone" for

failure as illustrated in Figure 11 (33). In the lower part of the figure are

plotted the temperature and stress distribution along the length of a turbine

bucket. The stress is highest at the base of the airfoil because the centri-

fugal loads imposed by total bucket airfoil length must be carried there. The

temperature, on the other hand, is purposely designed to be highest near the

tip, where the stresses are the lowest. At every point along the airfoil

length there is a specific centrifugal stress and temperature for which an

expected stress-rupture life can be calculated, resulting in the top curve.

At the base of the airfoil the life is very long as a result of the low tem-

perature, even though the stress is very high. At the tip the temperature is

high but the stress is very low, so again there is a very long life. Somewhere in the vicinity of the middle of the bucket airfoil length there is a combination of stress and temperature that results in the lowest computed life for a turbine bucket. The main point of Figure 11 is that from a strength viewpoint, the critical bucket temperature is not necessarily the highest temperature nor is the critical stress the highest stress. We are therefore interested in the typical value of stress at this limiting "critical zone." From a survey of several engines, we find that the stress level in this zone of the first stage buckets is generally between 15,000 and 20,000 psi. In addition to the stress from centrifugal force, the rotating bucket suffers from exposure to mechanical fatigue, thermal fatigue, and erosion and corrosion (33). All these factors will tend to reduce the life from that predicted from a consideration of stress-rupture alone - and candidate materials should have good resistance to failure from these causes. Initial comparisons are necessarily and conveniently made on the basis of only stress-rupture, however, and this approach will be used herein.

The stationary nozzle vanes (Fig. 12) are exposed to much lower stresses than the turbine buckets. Usual failure mechanisms are excessive bowing, local distortion and cracking, and erosion or corrosion (34). The bowing results from inability to withstand the creep conditions imposed by the bending loads of the high-velocity hot gases as the vanes redirect the flow of these gases toward the rotating buckets. The local distortion and cracking result from conditions of thermal fatigue.

Generally, the initial screening of vane materials may be made on the basis of stress-rupture strength (preferably creep strength) as a measure of ability to withstand the gas loads causing bowing. Thus we may examine candidate vane materials to withstand stresses, typically, in the range of 2500 to 7000 psi. The necessity for good thermal fatigue and corrosion resistance should not be minimized however.

To obtain a view of the potential of the available oxide-dispersion nickel materials as well as the other high temperature materials for buckets and vanes, these stress bands for buckets and vanes have been placed on the strength plots for these materials (Fig. 13). As mentioned the stress range of interest for buckets was 15,000 to 20,000 psi for superalloys. If we consider alternate materials of greater density, their strengths must be proportionately higher because the centrifugal stress will be higher. Thus it is necessary when considering other materials to convert the bucket stress range of 15,000 to 20,000 psi for nickel-base alloys to an equivalent required strength-density ratio of 52,500 to 70,000 inches. This is the band shown on the figure as "bucket stresses." Vanes are stationary parts and stresses are not related to density but because increased density means heavier engines, the heavier materials have been penalized (somewhat arbitrarily) by again converting the vane stress band of 2500 to 7000 psi to strength-density of 8,750 to 24,500 inches. This band is shown on the figure as "vane stresses."

Let us first consider turbine buckets. Considering the curve shown for an outstanding superalloy, as representative of this class of material, the superalloys are suitable for use at a maximum of about 1800° F for

1000 hour life. The strengths of the refractory metals are attractive, but as described earlier, none of the high strength alloys shown here may be used because their oxidation resistance is too poor.

An important point is made here for the oxide-dispersion-strengthened TD-nickel and DS-nickel (that are a basis for more oxidation resistant materials). They are shown to have too low strength at temperatures of interest. Clearly, if these materials are to find application in the rotating turbine buckets, it is necessary to increase the strength levels. It can be shown that it is also desirable to increase the slope of the strength-temperature curve and the question of additivity of strengthening mechanisms arises again. DuPont researchers have recently investigated solid-solution strengthening by adding molybdenum to the nickel matrix of TD-Ni. The product is called TD-NiM (31). The results (Fig. 14) indicate that the strength of TD-Ni has been increased markedly at low temperatures, but strengths at 1900° F and above are slightly inferior on a strength-to-density basis to TD-Ni and TD-NiM apparently does not have sufficient strength to replace nickel-base alloys in buckets. A sought after goal of a general elevation of the properties at all temperatures has not been achieved.

Let us now consider the stress levels for the stator vane. For this application the strength advantage of the dispersion-strengthened nickel materials indicates a potential for significantly increased operating temperature over the conventional superalloys. Indeed, DuPont's TD-nickel is under test for this application by engine manufacturers (35 and 36). In addition to this strength advantage, these materials do not have low

melting minor phases but retain the melting point of unalloyed nickel;
they do not suffer the microstructural damage (e.g., eutectic melting)
from momentary overtemperatures that may occur in conventional superalloys.
Although the above factors are favorable for dispersion-strengthened
nickel, other problems are posed as will be discussed later.

For the hottest sheet components of the turbine engines such as com-
bustion chambers and afterburner liners, it is difficult to give stress
levels. The major stresses and thus component failures are primarily the
result of thermal gradients (thermal fatigue) and, to a lesser degree,
vibration (mechanical fatigue). The materials may be exposed to very high
temperatures because of their position close to the combustion. Their
actual temperatures depend upon the ingenuity and good fortune of the
designer who attempts to have cooling air at the right place at the right
time under all engine operating conditions. He thus seeks to use the ma-
terial with the maximum temperature capability in regard to thermal
fatigue, mechanical fatigue, and oxidation resistance. The strength level
of the new dispersion-strengthened nickel and cobalt materials is adequate
to place them in contention for these applications.

As stated earlier, a primary failure mode of these sheet components
as well as nozzle vanes and, to a lesser degree, buckets is thermal
fatigue. The stress introduced in a part by changing temperature is di-
rectly proportional to the modulus of elasticity and the coefficient of
thermal expansion and inversely proportional to thermal conductivity.
Comparing dispersion-strengthened nickel to superalloys, their modulus
and coefficient of expansion are similar, but the coductivity of

dispersion-strengthened nickel is much higher (600 against 60 to

80 $Btu/ft^2/in./hr/^oF$), suggesting that induced thermal stresses would be

less as a result of lower thermal gradients. The ability of alloys to

withstand repeated thermal stresses that cause plastic yielding has been

shown by Manson and Coffin (37 and 38) in some cases to be related to the

tensile ductility (R/A) of the material in the temperature range of in-

terest. At 1600° to 1800° F, the tensile ductility (elongation) of TD-

Ni and TD-NiM are reported to be about 6 and 10 percent, respectively, as

compared to about 10 to 30 percent for a wrought nickel-base alloy

(Udimet 700). This latter comparison would not favor TD nickel. However,

in applications where a large number of cycles must be withstood, strength

becomes an important factor (39). The stability and strength of the dis-

persion strengthened materials should be to their advantage. Definitive

comparative data are needed under a variety of simulated service condi-

tions. Such data have not been available to the authors to date.

Additional problems in application of dispersion strengthened ma-

terials can be suggested based on a consideration of the fundamental

characteristics of these materials. Most obvious are welding problems.

The usual practice in joining of sheet components of engines is to weld.

Fusion, however, destroys cold-work, permits dispersoid agglomeration,

and the high strength is lost. (In some applications doublers may be

used to increase load carrying area in the joint, thus compensating for

loss in strength.) Diffusion bonding and brazing have been and are being

explored in depth; problems involving incipient recrystallization, de-

lamination of sheet and interdiffusion with brazes will have to be overcome.

This subject will be treated in later papers of this symposium, and we shall only emphasize its importance here.

Another problem is that the amount and kind of cold-work (or more generally the detailed thermomechanical processing history) in these dispersion-strengthened materials may be critical. Generally, wrought materials such as the dispersion strengthened materials are forged into the complex turbine-bucket shapes. If one produces bar stock with controlled and critical cold work, uniform through the cross section, to achieve optimum bar stock strength, the conversion of this round stock into a complex bucket shape will result in large variations in work and thus large variations in strength properties throughout the bucket shape.

Hypersonic Vehicles

Hypersonic vehicles provide another range of possible applications where the potential combination of strength, ductility, and oxidation resistance above 1800° F offered by dispersion strengthened materials might prove to be useful. Included are high lift-to-drag ratio (L/D) reentry vehicles, hypersonic aircraft, and reusable launch vehicles. In all of these, aerodynamic heating generates very high temperatures. This discussion will consider only the high L/D reentry vehicle. Although the quantitative nature of the materials properties necessary for these uses will, of course, vary with the specific type of vehicle and mission, the qualitative nature of the discussion will be generally applicable.

High L/D in a reentry vehicle provides the capability of landing on a runway, at a predetermined location or set of locations, independent of the time and location in orbit at which the decision to reenter is made.

A model of a typical projected vehicle is shown in Figure 15 (40). These
vehicles function as a glider for much of their trajectory and may or may
not have a jet engine to assist in final landing maneuvers. The trajec-
tories flown by such vehicles during reentry involve lower heat fluxes
but much longer times than the ballistic capsules used in past and current
manned space exploration. The total heat load is greater than for the
ballistic capsule. For L/D greater than 2, heat sink, ablative, or
convective-cooling thermal protection systems become very costly in terms
of the weight required, and the penalty is prohibitive for all except
small hot spots such as at a nose tip. The bulk of the heat must be
reradiated back to the environment by allowing the temperature of the
outer skin to increase to where this can be an effective form of heat re-
jection. Temperatures of over 2000° F in large areas and over 3000° F in
restricted areas can be encountered. It is possible to alleviate this
situation somewhat through a variable geometry design wherein a low L/D
configuration is used for the initial high thermal load portion of the
reentry and a high L/D configuration (e.g., by extending wings) is created
for lower altitude maneuvering and landing. For the purposes of this
discussion we will restrict ourselves to a fixed geometry vehicle, and a
typical temperature profile which can be reached is also shown in
Figure 15.

Two structural design approaches have been explored to cope with these
temperatures. In one no attempt is made to provide thermal protection
other than the normal heat sink and heat throughput characteristics of
the structure, and radiation from the hot surface. This was the approach

taken in the X15 and is illustrated in Figure 16. Although the thermal loads and temperatures were appreciably below those considered here, it was necessary to construct a superalloy hot structure (41). The over-all structure resembles that of a conventional aircraft; significant problems in coping with the thermal stresses generated by large variations in temperature in different parts of the structure had to be overcome by new structural design details.

The other general design approach makes use of a lower temperature load bearing structure insulated from a nonstructural outer heat shield which is permitted to reach very high temperatures and thus radiate away much of the aerodynamic heat input (Fig. 17) (42). Conventional alloys can be used for the low temperature structure. The outer radiative heat shield is usually of segmented panel construction to help accommodate thermal stresses. It generally must be of stiffened construction to cope with panel flutter and typical types of construction are also shown in Figure 17 with indications of appropriate fabrication methods. The strengths of metals which might be used for constructing such hot structures or heat shields are shown in Figure 18. Conventional nickel or cobalt base alloys (43) become inefficient above 1800° F and attention in the past has shifted to columbium and molybdenum alloys. The columbium alloy, Cb 752 (44) is a relatively ductile, fabricable, stable material from which reliable structural configurations could be made. As the strength of columbium alloys increase and molybdenum alloys such at TZM (45) are introduced, a very significant penalty in reduced weldability, and some penalty in fabricability and ductility is paid. There has been much

research towards improving this situation, but an even more significant factor in the use of these refractory metals is, of course, that currently available alloys require coatings to protect them from catastrophic oxidation. Major progress has been made in the development of a suitable coating technology and the feasibility of one flight performance has been demonstrated on the ASSET vehicle (17). Although the prospects for multimission reusability and reliability in coated refractory metals appear quite good, this will not be an easily attained technology and the possibilities of using dispersion-strengthened relatively oxidation-resistant nickel, iron or cobalt base materials warrants consideration. Thus, the yield strength of TD nickel sheet (46) is also shown in Figure 18.

For a hot structure design approach, tensile yield strength provides only a very preliminary evaluation; a better criterion would be the stress for 0.5 percent creep in X hours, where X is a function of the expected lifetime of the vehicle and might be of the order of 100 to 500 hours at temperature. However, using yield strength as only a "first-look" parameter for convenience in this discussion, a "bare minimum" requirement would be 20,000 psi and much higher values will probably be desired. Current dispersion strengthened nickel or cobalt sheet materials do not even begin to approach this level at temperatures of interest.

In the radiative heat shield structural design approach the outer heat shield itself, being nonstructural, requires considerably less strength than is necessary for a hot structure. Here the most significant loading is usually the transitory thermal stress that occurs during heat up and cooling portions of the trajectory. The thermal stress and thus

the strength required is very difficult to define. It is a complex function of the detailed design of the stiffened radiation shield and its attachment to the underbody. We suggest that a reasonable current goal is a yield strength of 10,000 psi at 2300° F. Usable heat shields could probably be built with lesser strengths, and higher strengths approaching those of the refractory materials which are now used for these purposes are obviously desired. For most flight paths and designs the air loading on the heat shield during the high temperature portion of the trajectory will be appreciably less than the possible thermal stress, but will be applied for longer times, and an 0.5 percent creep strength criterion will be applicable. Also, during low temperature portions of the trajectory the stresses may be considerably greater. Both of these will vary from application to application and extensive information will eventually be required for potential candidate materials to insure that they meet the short-time high temperature requirement, and also provide sufficient long-time and low temperature strengths. The contour lines and shading chosen for display on Figure 15 illustrate how much of a typical vehicle might be converted from a coated refractory metal to relatively oxidation resistant dispersion hardened metals if the latter can provide adequate heat shield properties in the 1800° to 2300° F range.

The subject of <u>relative</u> oxidation resistant materials must now be considered. Clearly, a dispersion strengthened alloy such as TD nickel must compete with coated columbium or molybdenum not only on the basis of strength but on the basis of the increased reliability offered by using a <u>relatively</u> oxidation resistant material. For the thin gages that

would be desired for an efficient stiffened heat shield construction and a multimission lifetime, unalloyed dispersion strengthened nickel would also require an oxidation resistant coating for 2300° F use. However, even unalloyed TD nickel will not oxidize as catastrophically as existing columbium or molybdenum structural alloys, the effect of coating flaws or failures should be significantly ameliorated, and it is reasonable to expect that it would be significantly easier to attain reliability with a coated dispersion strengthened nickel heat shield than a coated re-fractory metal heat shield. Dispersion strengthening of nickel alloys such as Nichrome can, of course, reduce or eliminate the need for a coating, and perhaps significantly increase reliability from an oxidation resistant standpoint. However, this will reduce the melting point and similarly the factor of safety for temperature overshoots due to emergency maneuvers or irregularities in the aerodynamic flow caused by local dis-tortion of the structure.

Two final aspects of the potential use of dispersion hardened nickel for radiative heat shields are worthy of note. One is the need to fabricate stiffened structures as shown in Figure 17, where joining is required. Of particular importance is the fact that in the region of the joint a thermal stress component may exist in the short transverse direction of the sheet, and delamination failure similar to that shown in Figure 19 for a current dispersion strengthened nickel is possible (47). Finally, the high thermal conductivity of dispersion - strengthened pure nickel relative to conventional nickel or refractory metal alloys may offer the

opportunity to reduce the magnitude of the thermal stresses in the heat shield, but introduces the problem of heat leakage through the support clip to the underlying structure.

Other Possibilities

Another set of application possibilities which makes use of the relatively unique combinations of properties offered by dispersion strengthening are for high temperature motors and inductor generators, thermoelectric and thermionic devices for power cables, in high temperature regions of supersonic or hypersonic aircraft, etc. The first requirement for such conductors will be chemical compatibility with the environment such as oxidation resistance in air. This can be met by appropriate selection of the metal to be used as a conductor, by alloying, or by appropriate coatings or claddings. Dispersion strengthening is then available as an option for providing the necessary strength at temperature or stability at the temperatures which might be required for fabrication, with minimum degradation of electrical conductivity or environmental stability (48). Figure 20 compares the elevated temperature strength of a commercially available dispersion strengthened copper (49) to the range for a wide variety of conventional copper alloys (50) and illustrates that dispersion strengthening can provide strength with little degradation from the electrical conductivity of unalloyed copper. Even so, it must compete on a cost-effectiveness basis with the approach of using high temperature alloys, a higher temperature metal, or a mechanically supported system. Finally, it is well to realize that even if a low-cost conductor

with adequate properties is provided by dispersion strengthening or a competitive route it may not see use. The even more difficult problems of providing adequate and compatible electrical insulation may dictate the use of a cooled rather than a hot electrical system.

A variety of other application possibilities (some already touched on) are worthy of mention. These include: ramjets, hot structures adjacent to the engines of supersonic aircraft; pumps or turbines for high temperature operation other than in air; advanced, regeneratively cooled rocket engines; magnetic materials for high temperature alternators; reactor fuel element cladding; and dimensionally stable materials for inertial guidance and optical systems.

Processing and Anisotropy

Several of the problems mentioned above appear to have a common root. As we have learned more about the high temperature and stability characterisitics of dispersion strengthened metals, it has become clear that a major contributor to these desirable characteristics is not only the dispersion itself but also the specific preferred structures introduced during processing of a material containing a dispersed phase and the retention of this favorable structure by presence of this unusually stable dispersed phase. The importance of such controlled processing and preferred structure is shown by published research, for example (32) and (51), and as with simple cold working, a significant anisotropy in properties can result. In fact, it can be expected that even more anisotropy (e.g., fibering) may result when working materials having a dispersed hard particle (52). The direction of working must be balanced

(or controlled) to avoid (or perhaps take advantage of) directional properties. Problems characteristic of such behavior have arisen several times during the preceding discussion of application possibilities, yet there is surprisingly little clarifying literature on the subject.

Doble and Quigg (53) have shown that in TD-Ni bar stock, elevated temperature strength is superior in the longitudinal direction compared to the transverse direction (Fig. 21); transverse strengths are shown to be as low as 1/3 to 1/4 the longitudinal strength. The marked anisotropy noted by these authors occurred only at elevated temperatures. Similar anistropy might be expected for worked dispersion-strengthened sheet, particularly in the short transverse or through-the-thickness direction unless carefully processed. This may lead to a tendency to delaminate when stressed in this direction by thermal stress or possibly during forming by bending, etc.

The over-all situation must be even more complex than would be suggested by simple concepts of cold work and fibering. The data of Doble and Quigg, in Figure 22, on tensile strength (at 2000° F) of TD-Ni as a function of bar stock diameter received from the producer and that of Fraser et al (32) on room temperature strength might be interpreted as due to the effects of increasing cold work. Thus, one might draw the conclusion from Figure 22 that material without the benefit of cold-work had an elevated temperature ultimate strength of near 8000 psi. Yet when Doble and Quigg worked a 1-inch bar an additional 50 percent by cold rolling, and heated it at 2200° F for 1 hour to produce a "coarse re-crystallized grain size," an ultimate tensile strength at 2000° F of

14,600 psi resulted. This was close to the strength of the initial 1-inch

bar but much greater than the 8000 psi attributed to the material that

had not been through the thermomechanical processing used to produce the

1-inch bar. Similar observations can be drawn from the data for TD-NiM

and TD NiC alloys reported by Barnett, Stuart, and Anders (31). The

authors report that either Cr or Mo additions to TD-Ni cause the worked

material to recrystallize at temperatures near 1800° F (1 hr) whereas

TD-Ni (similarly processed) does not recrystallize even at 2500° F.

They conclude that these materials (particularly TD-NiM) exhibit good

strength properties, however, indicating that "a lack of recrystallization

is not a necessary requirement for high strength in a dispersion hardened

product."

We need to know more about the mechanism by which processing in-

fluences the strength and anisotropy of these materials and more about

the effect of post-work annealing on strength. This will require

characterizing the resulting microstructures over the whole dimensional

range, from those visible in optical microscopy to those requiring trans-

mission electron microscopy. The practical implications of such influences

with respect to such problems mentioned previously as optimizing the

properties of complex shape forgings, coping with the multiaxial stress

systems, and the behavior of lap joints are obvious and very important.

Ductility in Oxide-Dispersion-Strengthened Metals

Historically as we have developed alloys with improved strength at

higher temperatures in a particular alloy system, we have been faced with

the unfortunate necessity, as a rule, to sacrifice ductility, particularly

at low temperatures. The question must be posed as to what price must we pay in ductility to achieve strength in dispersion-strengthened metals? One observation in this regard is that the tensile ductility in the more advanced oxide-dispersion strengthened nickel materials exhibits a decreasing trend with increasing temperature (for example, see ref. 1), even to very high temperatures where the ductility of melted alloys turns upward to very large values (as usable strength is lost).

Considering the strengthening mechanisms applied to dispersion materials, strength increases achieved by thermomechanical working do not necessarily lead to proportionate decreases in ductility (51 and 53) and surely there is opportunity for optimization of this trade-off during processing. On the other hand, another route to increased strength in dispersion strengthened materials is that of increasing the volume percent of the phase. Apparently this is usually achieved at the expense of reduced ductility as suggested by studies with early nickel materials (54), aluminum (55) and silver (56). Illustrative data for aluminum are shown in Figure 23. Definitive data for this trade-off for advanced dispersion-strengthened high temperature systems have apparently not been published and are needed.

The alloy developer asks the question - what is the minimum ductility that is acceptable? It would greatly simplify his problem if the answer could be given in terms of a minimum allowable reduction of area (or elongation) in a simple tensile test. Unfortunately, as we seek to use materials of lowest allowable ductility we have learned that we must be more sophisticated in this answer and relate ductility minimums in terms

of the specific property influenced by ductility rather than merely by a measure of ductility in a simple tensile test. Thus, in high strength steels, an important property measurement is fracture toughness to indicate propensity toward catastrophic brittle fracture when the material contains cracks. For turbine buckets, vanes, and sheet components, important ductility-related properties are impact strength to resist foreign-object damage, low notch-sensitivity for "fir-tree" attachment of buckets to disks, thermal and mechanical fatigue resistance, and formability. Frequently, in buckets, cracks initiated by thermal fatigue or mechanical fatigue propagate by creep and sensitivity to crack propagation in creep-rupture may be an important parameter. In creep-rupture, a specific minimum ductility at rupture would not be specified, but there is a preference toward a material with pronounced third stage creep so that measurement of deformation in service may be a criterion for warning of impending component failure.

As dispersion strengthened materials are developed that are candidates for application to hardware, more information will be needed on strength-ductility trade-offs in terms of the specific ductility-related properties of interest. Some of these data are being reported for TD-Ni (35) with encouraging results. Specific information on these trade-offs as a function of the structural parameters such as volume percent oxide, particle size and spacing, and thermomechanical processing are needed.

GENERAL COMMENTS

The objective of our discussion has been to analyze potential areas of applicability of dispersion strengthened materials and to suggest problems that must be resolved and information that must be acquired to permit such eventual use. An analysis of the current state of the science underlying these materials is the responsibility of the following papers and will not be attempted here. However, we should like to suggest some general attitudes or strategies which appear appropriate. It will be convenient to do this by modifying and expanding a conceptual framework suggested by Buessem (57) for ceramic research into that shown in Figure 24.

Much early and still existing development work correlates laboratory measured properties with process conditions such as a sintering temperature or extrusion ratio. Expanding work into the physical metallurgy of dispersion strengthened materials is concerned with identifying and characterizing the structure of these materials and correlating properties with structure. It is well recognized, of course, that intelligent and efficient development activities should make maximum use of information on structure-property relations even if it is only in terms of qualitative trends rather than quantitative predictions. Insertion of knowledge of structure between preparative conditions and properties is what converts an art to a science. It seems reasonably clear that in the immediate future, attention should be focused on fine dispersions with particle sizes less than 0.1 micron and spacings less than 1 micron, and that one of the first screening evaluations in a given composition or process should be stability of the dispersion in the

temperature range of interest. (Impurities can lead to particle ag-
glomeration and structural instability that can be revealed by very high
temperature anneals, refs. 51, 58, 59.) Effective development, evaluation,
and application of new materials also requires, as has been emphasized
throughout the paper, the recognition that the correlation between simple
laboratory measured properties and service use experience is not nec-
essarily obvious. For example, fracture propensity due to delamination
or stress corrosion may be a limiting factor in service and this may be
only weakly correlated, if at all, with tensile strength as the latter is
normally measured. One must beware of indiscriminate application to
dispersion strengthened materials, of properties or behavior criteria
which were adequate to predict the performance of conventional materials.

It is also reasonably clear that the properties attainable in even
pure metal-oxide particle systems will be a function of the precise de-
tails of the entire thermomechanical processing history. (This is not to
say, however, that similar properties cannot be obtained by markedly dif-
ferent methods of preparing dispersions.) A broad variety of structures
involving both the matrix and the dispersoid and appropriate interactions
can be attained, and characterizing these on both the microscopic and sub-
microscopic level will not be easy. To assist in achieving understanding
of the complex behavior trends which will result and to provide a base
for the eventual production of optimized materials will require particu-
lar emphasis in the area of synthesis or controlled preparation of de-
sired structures. The ability to synthesize microstructures with discreet
and controlled variations for use as experimental materials will, to a

considerable extent, determine the rate at which unequivocal trends can be identified and progress made in elucidating structure-property relations. A very sophisticated synthesis capability will also be needed to support the development of materials which attempt to combine dispersion strengthening with other strengthening mechanisms. Subtle interactions between both the alloy constituents and the thermomechanical processing appropriate to the various strengthening mechanisms can be expected. In fact, it is not inappropriate to suggest that in this area of metallurgy as in many others, there is the need for the specialist in synthesis, akin to the synthetic chemist, who is primarily interested in providing a wide variety of tools for attaining specified types of structure rather than doing this as an occasional by-product of studying structure-property relations or developing useful materials.

Recognizing these concepts it would seem imperative that in reporting research results on these materials, we use care in:

(a) Describing the precise details of all the processing and composition (including impurities) of the experimental specimens which were examined and, particularly, the details of the thermomechanical history.

(b) Describing the orientation relations among the processing history, the volume of the material actually examined in the specimen, and the direction of the examination or testing.

(c) Relating the observed behavior to structure on <u>both</u> the sub-optical and optical scale.

CONCLUDING REMARKS

A comparison of the presently understood characteristics of dispersion-strengthened materials with the general needs in high temperature materials leads to the following observations.

To date oxide-dispersion-strengthened materials have demonstrated retention of strength to higher temperatures than other strengthening mechanisms and show strength advantages at temperatures greater than 0.6 to 0.7 of the melting point of the matrix metal. This observation would suggest their development whenever it is necessary to extend the use of a particular alloy system or matrix to a higher temperature than can be achieved by conventional strengthening methods. If development is to lead to successful and rewarding application to hardware, however, it is obviously imperative that the development be conducted where the product may fill a need not readily met by an alternative material system or approach. This view emphasizes the opportunity for dispersion strengthening of oxidation resistant matrices (and some have been suggested) for use in the temperature range above 1800° F where alternate materials are not available.

The materials synthesized to date from an oxidation-resistant matrix are of interest for sheet applications in combustor and afterburner hardware and nozzle inlet guide vanes of propulsion systems and in heat shields of hypersonic or reentry aircraft. If their general strength level could be increased, these materials would be of considerable interest for much additional hardware such as rotating turbine buckets of turbine engines and as hot structure of hypersonic vehicles.

Many other opportunities will be identified where because of a specific property of a matrix, there is need to extend its use temperature. Specific examples are dispersion strengthening of aluminum (as has been done), because of its corrosion resistance and nuclear properties for fuel element cladding; of copper, because of its conductivity; and of tungsten, to provide, perhaps, the ultimate in use temperature in a metal-alloy system. The length of this list depends on the ingenuity and foresight of the developer and consumer; the authors claim no unique powers of prophecy or foresight.

With the recognition that the strengthening in many of these materials results from the interplay of thermomechanical processing and a stable dispersed particle of fine structural geometry has come the need for more basic understanding of the mechanism of this strengthening and for careful development programs to optimize properties in mill products and fabricated hardware. The use <u>at</u> <u>high</u> <u>temperature</u> of products containing controlled retained processing history may add a new dimension to high temperature alloy application. New problems are encountered in forming and joining and in relation to ductility and anisotropy.

Carefully conducted well-planned research and development will be required if we are to successfully synthesize, process, and apply this new class of materials.

Extending the scope of application of dispersion strengthening beyond that described above would, in large measure, depend upon development of low cost processes and/or determining that dispersion strengthening can be additive to other strengthening mechanisms over a broad temperature range.

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DISPERSION-STRENGTHENED ALLOYS
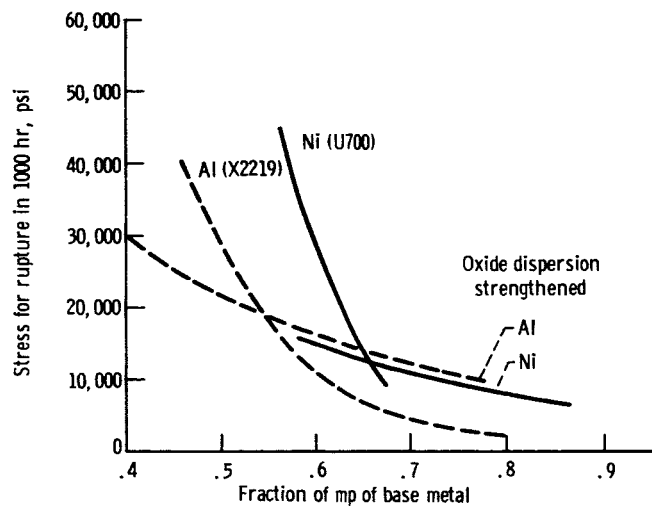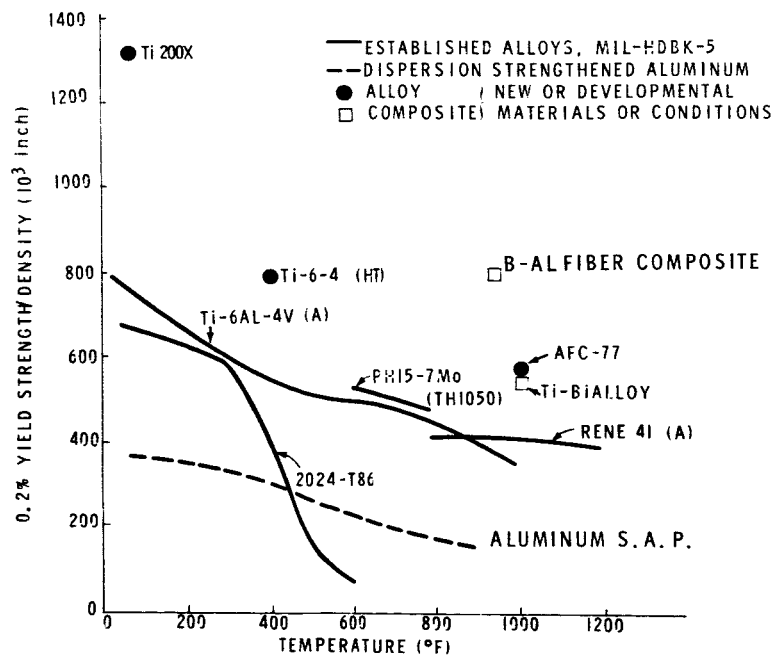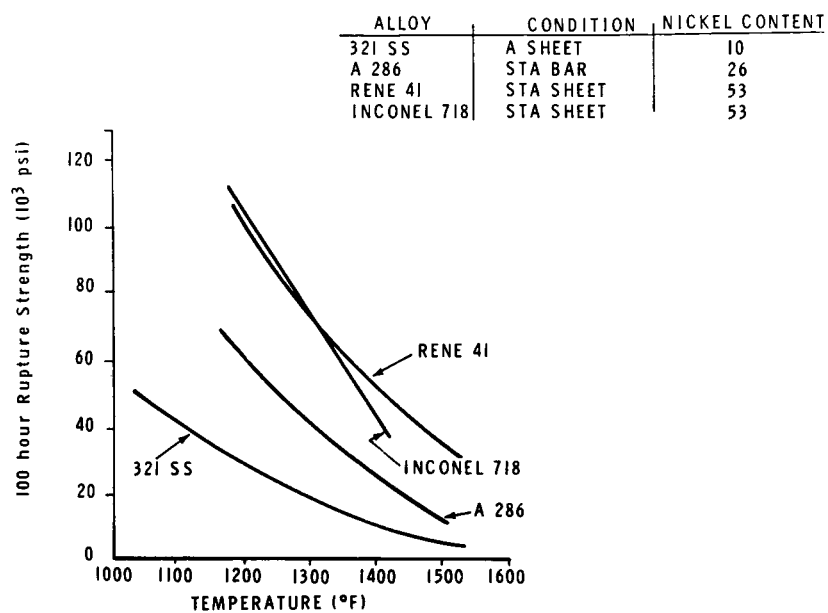## RETAIN STRENGTH TO HIGHER TEMPERATURES
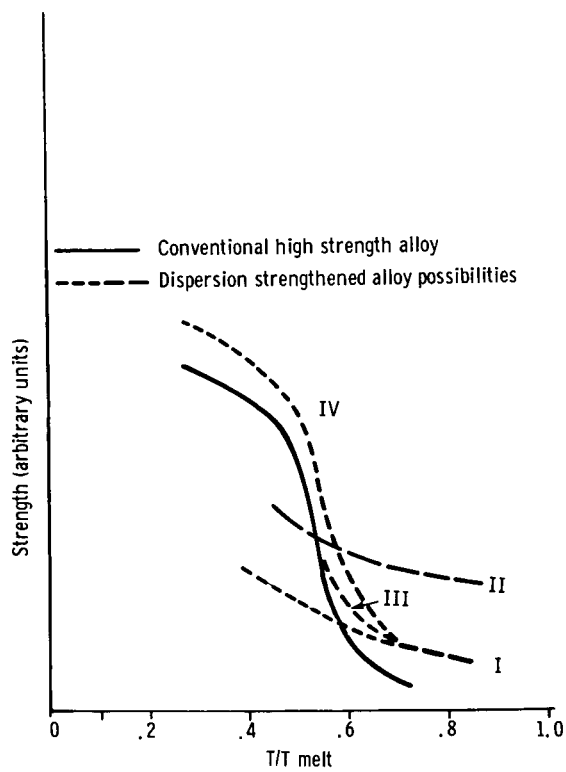

Figure 1.

CS-39967

Figure 2.

ALLOY	CONDITION	NICKEL CONTENT
321 SS	A SHEET	10
A 286	STA BAR	26
RENE 41	STA SHEET	53
INCONEL 718	STA SHEET	53

Figure 3.

Conventional high strength alloy

Dispersion strengthened alloy possibilities

Figure 4.

STRESS RUPTURE STRENGTHS OF SUPERALLOYS, CHROMIUM ALLOYS, AND REFRACTORY METAL ALLOYS
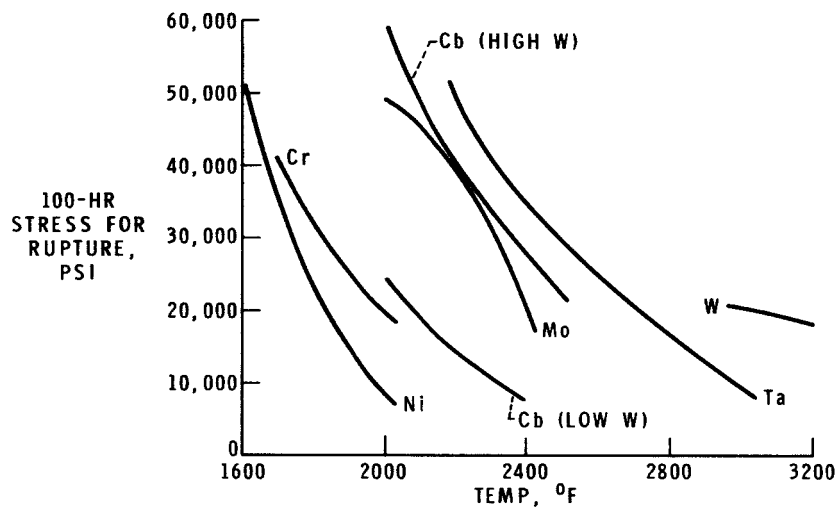
Figure 5.

STRENGTH OF BEST OXIDE-DISPERSION-STRENGTHENED TUNGSTEN
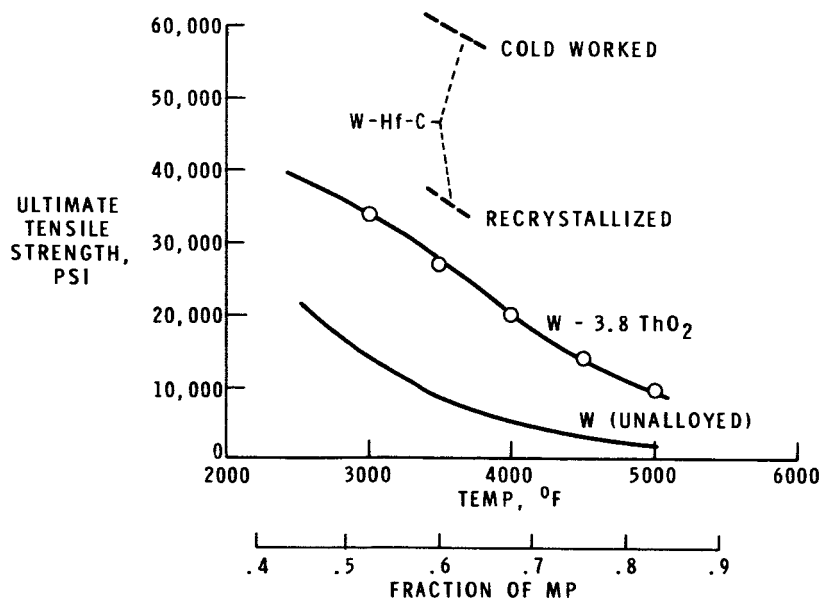
Figure 6.

OXIDATION OF REFRACTORY METALS

RATE OF WEIGHT CHANGE, (IN 1 HR), $\mu g/CM^2/HR$

TEMP, $^\circ F$
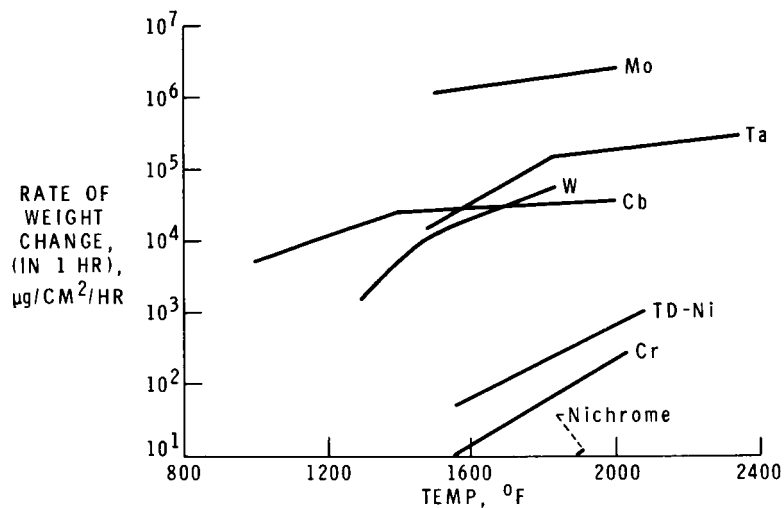
Figure 7.

CS-39962

OXIDATION RESISTANCE OF SEVERAL MATERIALS

TOTAL WEIGHT GAIN IN 100 HR, MG/CM^2

TEMP, $^\circ F$
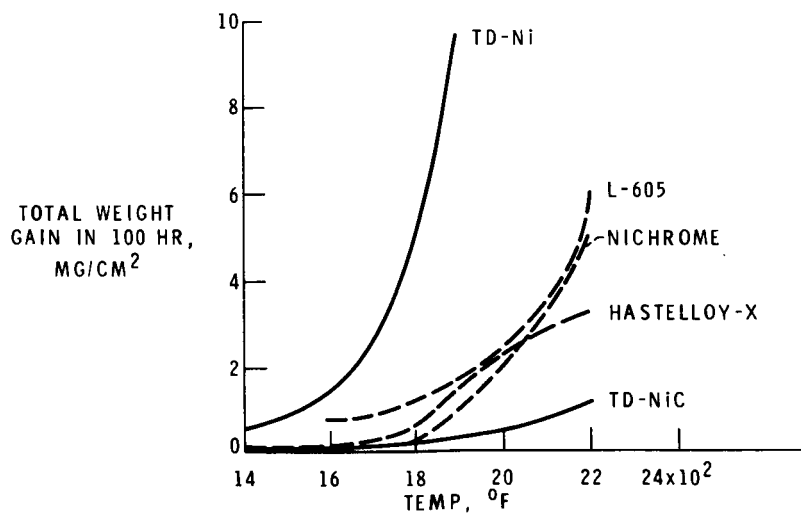
Figure 8.

CS-39966

DISPERSION STRENGTHENED Ni COMPARED TO
OTHER HIGH TEMPERATURE ALLOYS
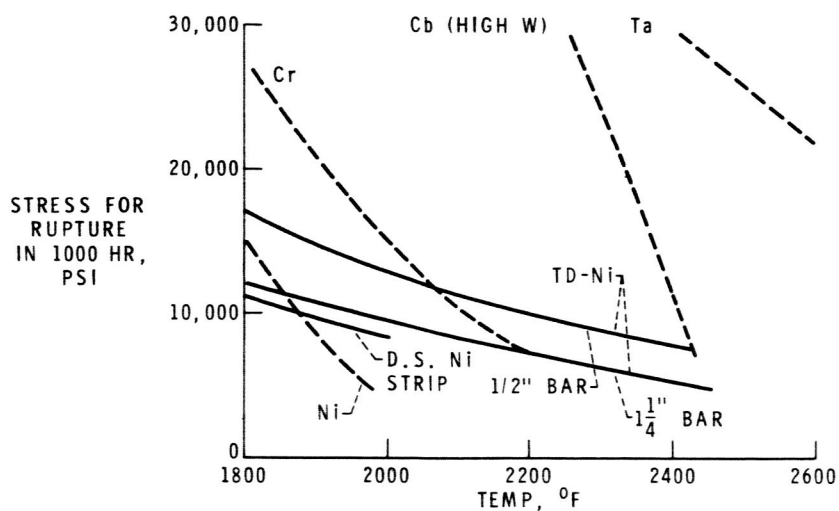
Figure 9.

CS-39965

TURBOJET ENGINE

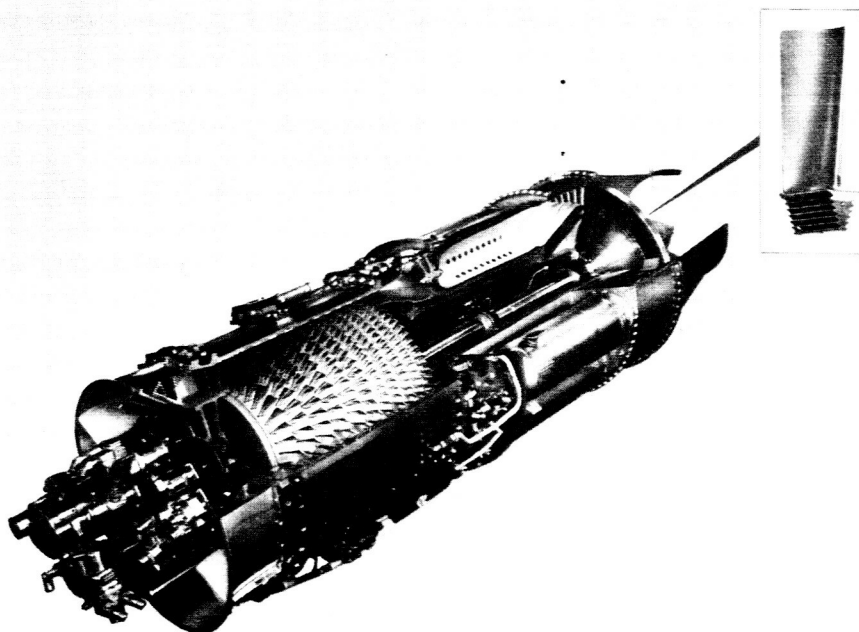CS-35979
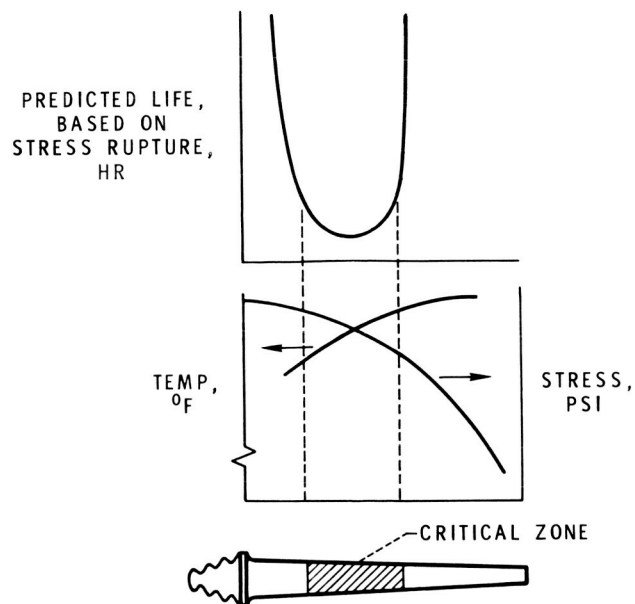
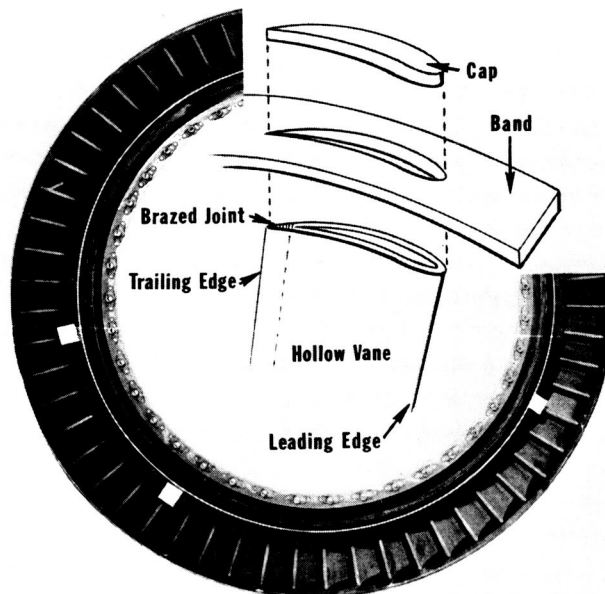Figure 10.

CRITICAL ZONE OF TURBINE BUCKETS

PREDICTED LIFE,
BASED ON
STRESS RUPTURE,
HR

TEMP,
°F

STRESS,
PSI

CRITICAL ZONE

Figure II.

NOZZLE VANE ASSEMBLY OF DISPERSION STRENGTHENED Ni

Cap

Band

Brazed Joint

Trailing Edge

Hollow Vane

Leading Edge

COURTESY OF G.E. CO.

Figure 12.

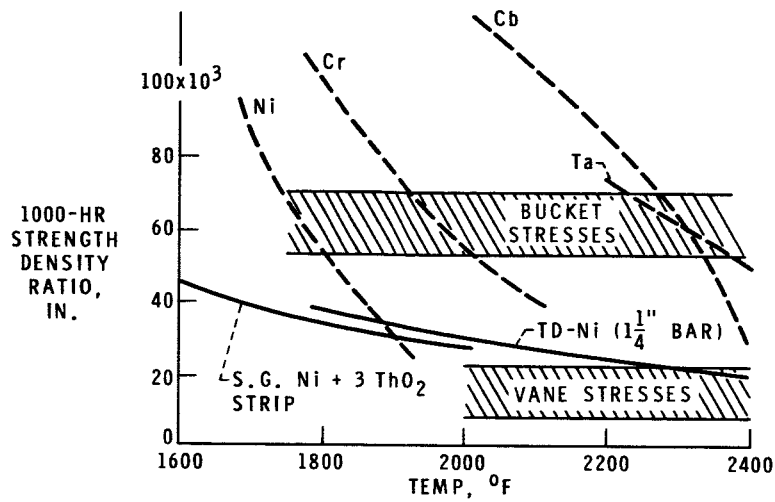## SUITABILITY OF MATERIAL STRENGTHS FOR
TURBOJET BUCKETS AND VANES

Figure 13.

EFFECT OF ALLOYING MATRIX ON STRENGTH OF
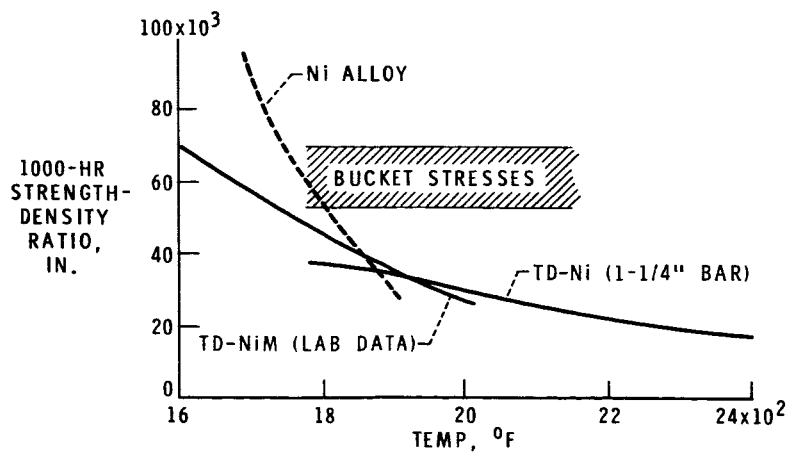## DISPERSION-STRENGTHENED NICKEL

Figure 14.

UNDERBODY
HEATSHIELDS ON A REENTRY GLIDER L/D-3

ABOVE 2300°F
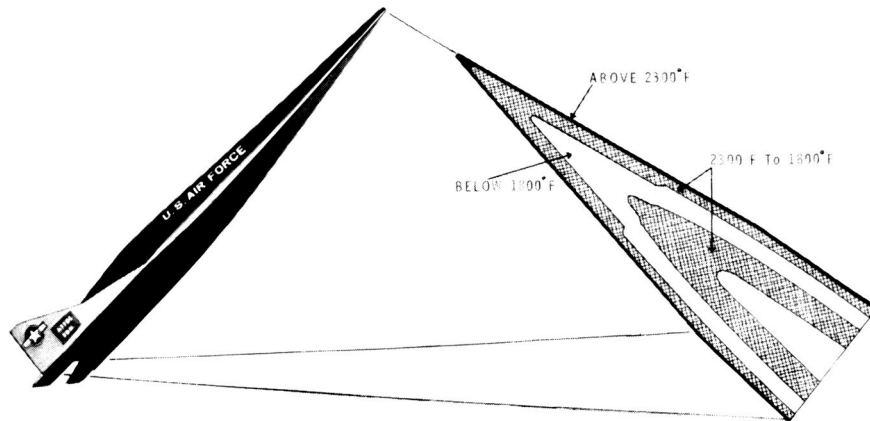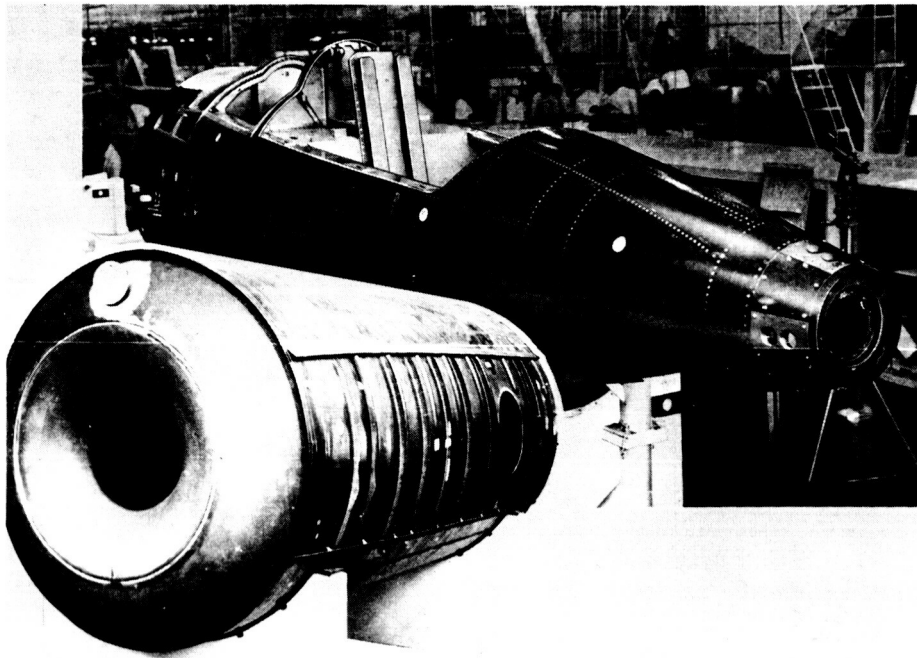
2300 F To 1800°F

BELOW 1800°F

U.S. AIR FORCE

Figure 15.

Figure 16.

RADIATION SHIELD CONFIGURATIONS
FOR HYPERSONIC AEROSPACE VEHICLES

Support Clip

Heat
Shield

Quartz
Insulation
Blanket

Superalloy
or Titanium
Load Bearing
Structure

HEAT SHIELD CONFIGURATION		
CONFIGURATION	CROSS SECTION	CONSTRUCTION
Integrally Stiffened		Machined or Extruded
Corrugated Stiffened		Spot or EB Weld Diffusion Bonded
Honeycomb Sandwich		Diffusion Bonded or Brazed
Truss-Core Sandwich		Spot or EB Weld Diffusion Bonded

Figure 17.

ALLOY	DENSITY(lb/in³)	APPROX. M.P. (°F)
MOLYBDENUM - TZM	0.37	4730
COLUMBIUM - Cb 752	0.33	4450
T.D. NICKEL SHEET	0.32	2600
NICKEL ALLOY R 41	0.30	2385

TZM

Cb 752

HOT STRUCTURE INTEREST

T.D. NICKEL

RADIATION SHIELD INTEREST

RENE 41

0.2% Y. S. (10³ psi)

TEMP. (°F)
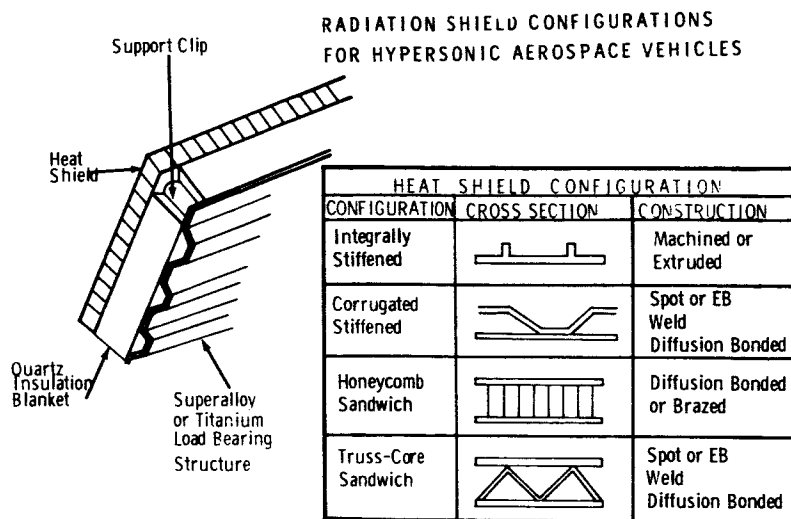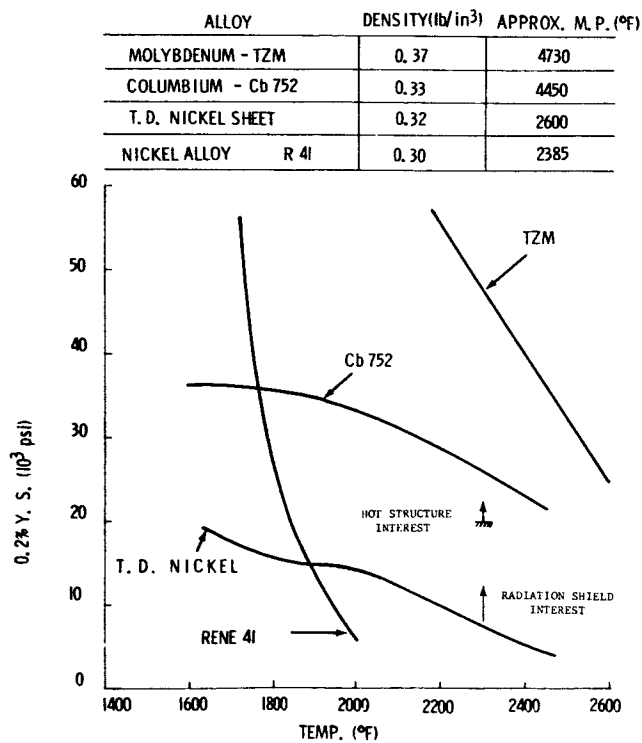
Figure 18.

Figure 19.

Figure 20.

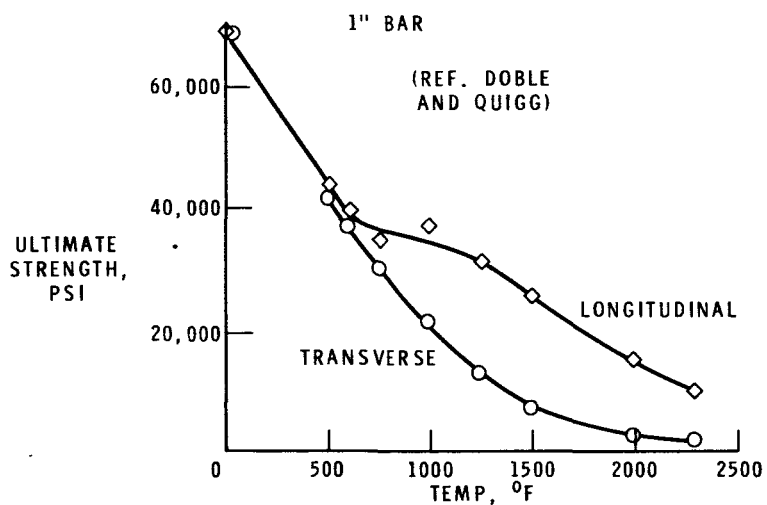LONGITUDINAL STRENGTH IS GREATER THAN
TRANSVERSE IN TD-Ni AT HIGH TEMPERATURE

1" BAR

60,000 —

(REF. DOBLE
AND QUIGG)

40,000 —

ULTIMATE
STRENGTH,
PSI

LONGITUDINAL

20,000 —

TRANSVERSE

0   500   1000   1500   2000   2500
TEMP, °F

Figure 21.

EFFECT OF COLD WORK ON 2000° F
TENSILE STRENGTH OF TD-Ni

20,000 —

(REF. DOBLE
AND QUIGG)

16,000 —

12,000 —

ULTIMATE
STRENGTH,
PSI

LONGITUDINAL

8,000 —

TRANSVERSE

4,000 —

0

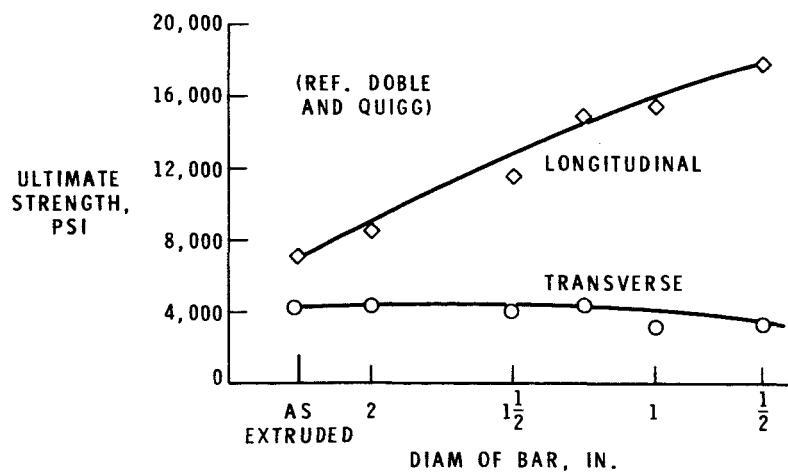AS   2   1½   1   ½
EXTRUDED

DIAM OF BAR, IN.

Figure 22.

INFLUENCE OF AMOUNT OF OXIDE ADDITION ON
STRENGTH AND DUCTILITY OF Al
ROOM TEMPERATURE

Figure 23.

SYNTHESIS⟷STRUCTURE⟷PROPERTIES⟷USE

Powder Blending	Dispersoid	Stability	Fracture
Coprecipitation	Size< 0.1µ	Anisotropy	•
Internal oxidation	Spacing< 1µ	•	
•		•	
•	Substructure	•	
•			
Sintering	Grain Size		
	and Shape		
Extruding	•		
	•		
Annealing	•		
	•		

Figure 24.